

CLAIMS

1. A method of forming a silica film coated on a substrate including the steps of:

producing a silica precursor formulation having a water content of no more than 5% by volume by adding silicic acid tetramethyl ester homopolymer to a solvent;

coating a substrate with the silica precursor formulation; and

curing the silica precursor formulation onto the substrate in a vaporous ammoniacal environment.
2. The method of claim 1 wherein the solvent is alcohol or an alcohol-aqueous solution.
3. The method of claim 1 wherein the silica precursor formulation contains an amount of tetramethoxysilane.
4. The method of claim 1 wherein the silica precursor formulation is formed by adding methyl-silicate-51 (MS-51), comprising >94% silicic acid tetramethyl ester homopolymer by volume, <3% tetramethoxysilane by volume and <3% methanol by volume, to the solvent.
5. The method of claim 1 wherein the silica precursor formulation comprises about 0.2-100 parts alcohol and 0.01-1 parts water for each part of MS-51.
6. The method of claim 1 wherein the silica precursor formulation comprises about 0.2 to 15 parts alcohol by volume and 0.01 to 0.1 part water by volume for each part of MS-51.
7. The method of claim 1 wherein the ratio of reagents in the silica precursor formulation is 1.0 part MS-51: 0.1 part water: 10.0 parts alcohol by volume.

8. The method of claim 1 wherein the coating is performed by spin coating or dipping.
9. The method of claim 1 wherein the coating further includes allowing the coating to settle before curing.
10. The method of claim 1 wherein the curing is carried out by placing the coated substrate in a closed ammoniacal environment.
11. The method of claim 10 wherein the ammoniacal environment contains water, ammonia and alcohol.
12. The method of claim 11 wherein the solvent used in the formation of the silica precursor is an alcohol, and the alcohol contained in the ammoniacal environment is the same alcohol as used in the formation of the silica precursor.
13. The method of claim 1 further including controlling the solvent content to control characteristics of the silica film.
14. The method of claim 1 further including controlling the alcohol content in the ammoniacal environment to control characteristics of the silica film.
15. The method of claim 1 further including controlling a pore size of the silica film by controlling the solvent content and type in the silica precursor formulation.
16. The method of claim 1 further including controlling a pore density of the silica film by controlling the solvent content and type in the ammoniacal environment.
17. The method of claim 12 further including controlling a porosity of the silica film by controlling the solvent content and type in the precursor formulation and alcohol content and type in the ammoniacal environment.

18. A silica precursor formulation comprising:
about 1 part by volume including >94% silicic acid tetramethyl ester homopolymer and <3% tetramethoxysilane;
about 0.01-100 parts by volume of an alcohol; and
about 0.01-1 part by volume water;
such that the water content is no more than 5% by volume.
19. A silica film having a refractive index between 1.1 and 1.56 and a film thickness less than 100 microns formed by a method including the steps of:
producing a silica precursor formulation having a water content of no more than 5% by volume by adding silicic acid tetramethyl ester homopolymer to a solvent;
coating a substrate with the silica precursor formulation; and
curing the silica precursor formulation onto the substrate in a vaporous ammoniacal environment.
20. The silica film of claim 19 having a thickness of less than 1 μm .
21. The silica film of claim 19 comprising a continuous, interconnected, nanoporous silica network.
22. The silica film of claim 19 comprising a hardness greater than 7H on pencil scale.
23. The silica film of claim 19 wherein the film is resistant to washing with water, alcohols, common acids and alkalis.
24. The silica film of claim 19 wherein the film is anti-fogging.
25. Use of the silica film formed by the method of claim 1 in a coating on a transparent substrate to provide an anti-reflective and/or anti-fogging and/or

protective coating

26. An anti-reflection coating for a transparent substrate comprised by a silica film formed according to the method of claim 1.

27. An anti-fogging coating for a transparent substrate comprised by a silica film formed according to the method of claim 1.

28. An anti-scratch coating for a substrate comprised by a silica film formed according to the method of claim 1.

29. An anti-static coating for a substrate comprised by a silica film formed according to the method of claim 1.

30. A method of forming a silica film coated on a substrate including the steps of:

producing a silica precursor formulation having a water content of no more than 5% by volume by adding silicic acid tetramethyl ester homopolymer to a solvent;

coating a substrate with the silica precursor formulation;

placing the coated substrate in a closed solvent environment;

establishing equilibrium between the solvent in the precursor formulation and the solvent environment; and

curing the silica precursor formulation onto the substrate in an ammoniacal environment containing solvent by introducing ammonia vapour and water vapour to the closed solvent environment.

AMENDED CLAIMS

[received by the International Bureau on 17 February 2005 (17.02.05);
original claims 1-28 replaced by new claims 1-34 (5 pages)]

1. A method of forming a silica film coated on a substrate including the steps of:
producing a silica precursor formulation by adding silicic acid tetramethyl
5 ester homopolymer to a solvent;
coating a substrate with the silica precursor formulation; and
curing the silica precursor formulation onto the substrate in an ammoniacal environment.
2. The method of claim 1 wherein the solvent is alcohol or an alcohol-
10 aqueous solution.
3. The method of claim 1 wherein the silica precursor formulation contains an amount of tetramethoxysilane.
4. The method of claim 1 wherein the silica precursor formulation is formed by adding methyl-silicate-51 (MS-51), comprising >94% silicic acid
15 tetramethyl ester homopolymer, <3% tetramethoxysilane and <3% methanol, to the solvent.
5. The method of claim 1 wherein the silica precursor formulation comprises about 0.01-1 parts water and 0.01-100 parts alcohol for each part of MS-51.
- 20 6. The method of claim 1 wherein the silica precursor formulation comprises about 0.01 to 15 parts alcohol and 0.01 to 1 part water for each part of MS-51.
7. The method of claim 1 wherein the ratio of reagents in the silica precursor formulation is 1.0 part MS-51: 0.1 part water: 10.0 parts alcohol.

8. The method of claim 1 wherein the coating is performed by spin coating or dipping.
9. The method of claim 1 wherein the coating further includes allowing the coating to settle before curing.
- 5 10. The method of claim 1 wherein the silica film comprises a continuous, interconnected, nano-porous silica network.
11. The method of claim 1 wherein the curing is carried out by placing the coated substrate in a closed ammoniacal environment.
12. The method of claim 11 wherein the ammoniacal environment
10 contains water, ammonia and alcohol.
13. The method of claim 12 wherein the solvent used in the formation of the silica precursor is an alcohol, and the alcohol contained in the ammoniacal environment is the same alcohol as used in the formation of the silica precursor.
- 15 14. The method of claim 1 further including controlling the solvent content to control characteristics of the silica film.
15. The method of claim 1 further including controlling the alcohol content in the ammoniacal environment to control characteristics of the silica film.
16. The method of claim 1 further including controlling a pore size of the
20 silica film by controlling the solvent content and type in the silica precursor formulation.
17. The method of claim 1 further including controlling a pore density of the silica film by controlling the solvent content and type in the ammoniacal environment.

18. The method of claim 12 further including controlling a porosity of the silica film by controlling the solvent content and type in the precursor formulation and alcohol content and type in the ammoniacal environment.

19. A method of forming a silica precursor formulation, including the steps of:

mixing silicic acid tetramethyl ester homopolymer with a solvent.

20. The method of claim 19 wherein the solvent is an alcohol or an alcohol-aqueous solution.

21. The method of claim 19 wherein the silica precursor formulation is formed by adding methyl-silicate-51 (MS-51), comprising >94% silicic acid tetramethyl ester homopolymer, <3% tetramethoxysilane and <3% methanol, to the solvent.

22. A silica precursor formulation comprising:
about 1 part including >94% silicic acid tetramethyl ester homopolymer and
<3% tetramethoxysilane;
about 0.01-100 parts of an alcohol; and
about 0.01-1 part water.

23. A silica film having a refractive index between 1.1 and 1.56 and a film thickness less than 100 microns formed by a method including the steps of:
producing a silica precursor formulation by adding silicic acid tetramethyl ester homopolymer to a solvent;
coating a substrate with the silica precursor formulation; and
curing the silica precursor formulation onto the substrate in an ammoniacal environment.

24. The silica film of claim 23 having a thickness of less than 1 μm .
25. The silica film of claim 23 comprising a continuous, interconnected, nano-porous silica network.
26. The silica film of claim 23 comprising a hardness greater than 7H on
5 pencil scale.
27. The silica film of claim 23 wherein the film is resistant to washing with water, alcohols, common acids and alkalis.
28. The silica film of claim 23 wherein the film is anti-fogging.
29. Use of the silica film formed by the method of claim 1 in a coating on a
10 transparent substrate to provide an anti-reflective and/or anti-fogging and/or protective coating
30. An anti-reflection coating for a transparent substrate comprised by a silica film formed according to the method of claim 1.
31. An anti-fogging coating for a transparent substrate comprised by a
15 silica film formed according to the method of claim 1.
32. An anti-scratch coating for a substrate comprised by a silica film formed according to the method of claim 1.
33. An anti-static coating for a substrate comprised by a silica film formed according to the method of claim 1.
- 20 34. A method of forming a silica film coated on a substrate including the steps of:
- producing a silica precursor formulation by adding silicic acid tetramethyl ester homopolymer to a solvent;
- coating a substrate with the silica precursor formulation;
- 25 placing the coated substrate in a closed solvent environment;

establishing equilibrium between the solvent in the precursor formulation and the solvent environment; and

- 5 curing the silica precursor formulation onto the substrate in an ammoniacal environment containing solvent by introducing ammonia vapour and water vapour to the closed solvent environment.